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(54) Title: GERMICIDAL COMPOSITION

(57) Abstract

This invention relates to a germicidal composition comprising (A) an inorganic peroxide, (B) an incomplete ester of a polyhydric alcohol with an organic acid, and (C) an alkali earth metal salt. The composition is excellent in sterilizing effects, long-lasting property thereof and also storage stability.

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DESCRIPTION

GERMICIDAL COMPOSITION

Technical Field

The present invention relates to germicidal compositions which can be used for a wide variety of applications ranging from applications to foods, paper and fibers to domestic applications. Specifically, the present invention relates to germicidal compositions which can be used typically for the sterilization and disinfection of city water for purification, the sterilization and disinfection of general foods led by meat and fishery products, the antisepsis of wood, antisepsis in paper manufacturing processes, and the sterilization and disinfection of toilet, bath rooms and kitchen at home and which are excellent in sterilizing effects and storage stability.

Background Art

It is known that an inorganic peroxide such as sodium perborate, sodium percarbonate or sodium persulfate generates both hydrogen peroxide and nascent oxygen when dissolved in water and owing to these hydrogen peroxide and nascent oxygen, the peroxide exhibits cleaning, bleaching, sterilizing and disinfecting effects.

Use of such an inorganic peroxide alone however results in release of oxygen at once in a short time so that oxygen will escape in the form of large bubbles to the outside of a system to which the inorganic peroxide is applied. Nascent oxygen will therefore be lost quickly, leading to the drawback that no sufficient sterilizing effects will be provided.

With a view toward overcoming such a drawback of inorganic peroxides, compositions have been developed in which an inorganic peroxide is mixed with, as an activator for the inorganic peroxide, an organic acid ester such as an acetate ester or a propionate ester (Japanese Patent Laid-Open Nos. 14886/1973, 25011/1977, 139500/1970 and 63504/1987). These compositions each forms an organic peracid when reacted with water at a place to be sterilized. The resultant organic peracid reacts further with water and generates oxygen, thereby exhibiting sterilizing effects.

These compositions however undergo such reactions in the presence of even a slightest amount of water so that when stored for a long time, they will give off an irritating organic acid odor. Among these compositions, those containing an inorganic peroxide and an ester of a polyhydric alcohol and an organic acid in combination are relatively stable against water (Japa-

nese Patent Laid-Open No. 25011/1977). They are, on the other hand, accompanied by the drawback that due to their low solubility in water, an organic peracid is not produced sufficiently, hence oxygen is formed only at a low rate and their sterilizing power is low.

Incidentally, compositions with an organic peracid incorporated therein as is are accompanied with the drawback that they have an irritating odor and are hence low in commercial value. Further, those composed of an organic peracid alone will involve the drawback that they decompose when stored for a long time.

Accordingly, an object of the present invention is to provide a germicidal composition which stably generates an organic peracid, has sustained sterilizing power and has excellent storage stability.

Disclosure of the Invention

The present invention provides a germicidal composition comprising the following components (A), (B) and (C):

(A) an inorganic peroxide,

(B) an incomplete ester of a polyhydric alcohol with an organic acid, and

(C) an alkali earth metal salt.

By the combination of these three components, the

resulting germicidal composition according to the present invention has good storage stability, good water solubility and high and long-lasting sterilizing effects so that it can be used for a wide variety of applications ranging from applications to foods, paper and fibers to domestic applications.

Best Modes for Carrying Out the Invention

Examples of the component (A), that is, the inorganic peroxide used in the present invention include sodium percarbonate, sodium perborate, sodium peroxytripolyphosphate, sodium peroxypyrophosphate and sodium peroxysilicate. Among them, sodium percarbonate and sodium perborate are particularly preferred.

The component (B) which is the incomplete ester of the polyhydric alcohol with the organic acid serves as an activator for the inorganic peroxide as the component (A). It reacts with the component (A) to form an organic peracid.

One of characteristic features of the present invention resides in the incorporation of the incomplete ester of the polyhydric alcohol with the organic acid as an activator for the inorganic peroxide. The composition with this incomplete ester incorporated therein has excellent water-solubility and dramatically im-

proved sterilizing effects compared with conventional compositions containing the complete ester of a polyhydric alcohol with an organic acid. The term "incomplete ester" as used herein means an ester in which the degree of esterification of a polyhydric alcohol is less than 100%. The esterification degree can preferably be 5-95%, with 60-95% being particularly preferred. Incidentally, the esterification degree can be measured from the degree of absorption at 3500 cm^{-1} in an infrared absorption spectrum.

Illustrative examples of the polyhydric alcohol can include glycerins such as glycerin, diglycerin, triglycerin and polyglycerin; alkali-modified sucroses such as sorbitol, glutitol, pentaerythritol, alkylpolyglycosides and alkylfuranosides; alkylene-oxide adducts of these glycerins and sucroses. Preferred examples of the organic acid can include saturated or unsaturated C_{1-8} fatty acids. Specific examples include acetic acid, propionic acid, butyric acid, valeric acid, caproic acid, enanthic acid, octanoic acid, acrylic acid, methacrylic acid, crotonic acid, allylacetic acid, dimethylacrylic acid and monocarboxylic acids. Dicarboxylic acids such as oxalic acid, malonic acid, succinic acid, maleic acid and fumaric acid can also be employed.

Examples of the alkali earth metal salt as the component (C) can include alkali earth metal salts of inorganic acids and halides of alkali earth metal salts. Illustrative alkali earth metals can include calcium and magnesium, illustrative inorganic acids can include sulfuric acid, nitric acid, phosphoric acid and carbonic acid, and illustrative halogens can include chlorine and bromine. Specific examples of the component (C) include magnesium sulfate, calcium sulfate, magnesium phosphate, calcium phosphate, magnesium nitrate, calcium nitrate, magnesium chloride, calcium chloride, basic magnesium carbonate and calcium carbonate, and anhydrous salts thereof.

The component (C) has stabilizing effects for the organic peracid to be formed through the reaction between the component (A) and the component (B), and the addition of the component (C) has improved the long-lasting ability of sterilizing effects.

In the germicidal composition according to the present invention, the components (A) to (C) can be mixed properly depending on its application purpose. In view of sterilizing effects, storage stability, economy and the like, it is preferred to mix 0.01-10 parts by weight of the component (B) and 0.01-90 parts by weight of the component (C) with 1 part by weight of

the component (A). Particularly preferred is to add the component (C) 0.01-100 times, particularly 0.1-20 times in weight relative to the component (B).

In addition to the above three components, the germicidal composition of the present invention can contain various additives such as surfactants, inorganic or organic alkali metal salts, builders, flavorants, pigments, dyes, pH regulators and metallic chelating agents. Illustrative surfactants can include nonionic, anionic and ampholytic ones. Specific examples can include nonionic surfactants such as polyoxyethylene (hereinafter abbreviated as "POE") C_{6-22} alkyl ethers, POE C_{4-18} alkylphenol ethers, block or random polyoxypropylene-polyoxyethylene alkyl ethers, POE phenylphenol ether, POE styrenated phenol ether and POE tribenzylphenol ether; anionic surfactants such as lignin sulfonate salts, alkylbenzene sulfonate salts, alkyl sulfonate salts, POE alkyl sulfonate salts, POE alkylphenylether sulfonate salts, POE alkylphenylether-phosphate ester salts, POE phenylphenolether sulfonate salts, POE phenylphenolether phosphate ester salts, naphthalene sulfonate salts, naphthalenesulfonic acid-formaldehyde condensates, POE tribenzylphenolethersulfonate salts and POE tribenzylphenylphenolether phosphate esters; and ampholytic surfactants such as

alkylaminotrimethylglycines, alkyldimethylamine oxides and alkyldiaminoethylglycine hydrochlorides. They may be used either singly or in combination. The content of the surfactant in the germicidal composition can be 0-20 wt.%, preferably 1-10 wt.%.

Preferred examples of the alkali metal salts include alkali metal salts of organic acids and those of inorganic acids. Specific examples of the former ones can include alkali metal salts of carboxylic acids such as succinic acid, malonic acid, citric acid and gluconic acid, glutaric acid; and those of the latter ones can include alkali metal salts of phosphoric acid compounds such as tripolyphosphoric acid, hexametaphosphoric acid and phosphoric acid, and alkali metal salts of mineral acids such as Na_2SO_4, K_2SO_4 and $NaHSO_4$. The addition of such a salt makes it possible to provide improved storage stability and also to prevent the generation of an organic peracid odor. These salts may be used either singly or in combination. These alkali metal salt can be added preferably in an amount of 0.1-10 wt.%, with 0.5-5 wt.% being particularly preferred.

Illustrative examples of the pH regulator include organic acids such as citric acid, malonic acid, succinic acid and gluconic acid. They may be used either singly or in combination. It is preferred to add such

a regulator in such an amount as providing an aqueous solution of the composition of this invention with pH 5-10, for example in an amount of 0.5-5 wt.%.

Examples of the metal chelating agent can include ethylenediaminetetraacetic acid, nitrilotriacetic acid, tripolyphosphoric acid and polyhydroxyacrylic acid, and salts thereof.

The germicidal composition according to the present invention can be produced in a solid form such as powder, granules or tablets by a method known *per se* in the art. Upon use, it is diluted with water and is then applied. The concentration of the composition differs with the item, place or the like to be treated. In general, however, 25-1,000 ppm is desired in terms of the concentration of the inorganic peroxide.

Alternatively, the components (A), (B) and (C) can be furnished in separate packages and upon use, they are diluted together with water and are then applied together.

Examples

The present invention will hereinafter be described more specifically by various examples. It should however be borne in mind that this invention is by no means limited to or by the examples.

Example 1

Various germicidal compositions were prepared as shown in Tables 1-3 to meet the requirements of various fields. After they were hydrated, the amounts of organic peracids released, that is, the concentrations of the organic peracids upon elapsed times of 15, 30 and 60 minutes after the hydration were measured, respectively. The results are shown in Tables 1-3.

Table 1

	(A) Inorganic peroxide	(B) Activator for the inorganic peroxide	(C) Alkali earth metal salt	(A)/(B)/(C) Concentration (ppm) upon hydration	Amount of peracetic acid (after hydration) (ppm) 15 min	30 min	60 min
Invention product	Sodium percarbonate	Glycerin monoacetate (esterification degree: 33.3%)	Magnesium sulfate	100/0/0	0	0	0
				100/100/0	350	500	50
				100/100/100	350	500	150
				100/100/500	350	400	150
				100/100/800	300	400	200
				100/100/1500	300	400	300
				100/0/800	0	0	0
				0/100/800	0	0	0
	Sodium percarbonate	Glycerin diacetate (esterification degree: 66.7%)	Magnesium sulfate	100/0/0	0	0	0
				100/100/0	400	600	50
				100/100/100	300	500	200
				100/100/500	300	500	200
				100/100/800	350	400	250
				100/100/1500	350	400	300
				100/0/800	0	0	0
				0/100/800	0	0	0
Comparative product	Sodium percarbonate	Glycerin triacetate (comparative product, esterification degree: 100%)	Magnesium sulfate	100/0/0	0	0	0
				100/100/0	170	120	30
				100/100/100	150	130	120
				100/100/500	150	170	160
				100/100/800	150	170	170
				100/100/1500	100	150	100
				100/0/800	0	0	0
				0/100/800	0	0	0

Table 2

	(A) Inorganic peroxide	(B) Activator for the inorganic peroxide	(C) Alkali earth metal salt	(A)/(B)/(C) Concentration (ppm) upon hydration	Amount of peracetic acid (after hydration)(ppm) 15 min	30 min	60 min
Invention product	Sodium percarbonate	Glucose triacetate (esterification degree: 60%)	Magnesium sulfate	100/0/0	0	0	0
				100/100/0	400	350	50
				100/100/100	400	550	300
				100/100/500	350	550	300
				100/100/800	350	500	450
				100/100/1500	350	500	450
				100/0/800	0	0	0
				0/100/800	0	0	0
	Sodium percarbonate	Glucose tetraacetate (esterification degree: 80%)	Magnesium sulfate	100/0/0	0	0	0
				100/100/0	500	300	50
				100/100/100	300	600	250
				100/100/500	300	600	350
				100/100/800	320	500	350
				100/100/1500	310	500	400
				100/0/800	0	0	0
				0/100/800	0	0	0
Comparative product	Sodium percarbonate	Glucose pentaacetate (comparative product, esterification degree: 100%)	Magnesium sulfate	100/0/0	0	0	0
				100/100/0	200	150	50
				100/100/100	150	130	75
				100/100/500	150	130	80
				100/100/800	100	100	100
				100/100/1500	50	100	150
				100/0/800	0	0	0
				0/100/800	0	0	0

Table 3

	(A) Inorganic peroxide	(B) Activator for the inorganic peroxide	(C) Alkali earth metal salt	(A)/(B)/(C) Concentration (ppm) upon hydration	Amount of propionic acid (after hydration) (ppm) 15 min	30 min	45 min
Invention product	Sodium perborate	Sucrose tetrapropanate (esterification degree: 50%)	Magnesium sulfate	100/0/0	0	0	0
				100/100/0	400	500	50
				100/100/100	350	500	350
				100/100/500	350	550	400
				100/100/800	350	550	400
				100/100/1500	300	600	500
				100/0/800	0	0	0
				0/100/800	0	0	0
	Sodium perborate	Sucrose hexapropanate (esterification degree: 75%)	Magnesium sulfate	100/0/0	0	0	0
				100/100/0	450	500	50
				100/100/100	400	500	400
				100/100/500	400	600	450
				100/100/800	400	600	450
				100/100/1500	350	650	550
				100/0/800	0	0	0
				0/100/800	0	0	0
Comparative product	Sodium perborate	Sucrose octapropanate (esterification degree: 100%)	Magnesium sulfate	100/0/0	0	0	0
				100/100/0	150	50	10
				100/100/100	100	50	50
				100/100/500	50	50	50
				100/100/800	50	50	50
				100/100/1500	10	50	50
				100/0/800	0	0	0
				0/100/800	0	0	0

As is apprent From Tables 1-3, it should be understood that each invention composition comprising the components (A), (B) and (C) generates a corresponding organic peracid in a large amount and furthermore, the organic peracid remains at a high concentration for a long time.

Example 2 Storage Stability Test

In accordance with the compositions shown in Table 4, germicidal compositions were prepared by proportioning the components. They were hermetically stored at 40°C and 75% humidity for a month. Each composition was then checked for any organic acid odor.

As is shown in Table 4, it has been found that, although each composition comprising the components (A), (B) and (C) has good storage stability, its storage stability can be improved further by the addition of an alkali metal salt.

Table 4

	(A) Inorganic peroxide	(B) Activator for the inorganic peroxide	(C) Alkali earth metal salt	Alkali metal salt	PH regulator, etc.	Organic acid odor at 40°C (75% RH)	
						Before storage	1 Month later
Invention product	Sodium percarbonate 5 wt.% 5 wt.%	Glycerin diacetate 5 wt.% 5 wt.%	Magnesium sulfate 49 wt.% 9 wt.%	Sodium sulfate 40 wt.% 80 wt.%	Citric acid 1 wt.% 1 wt.%	 No odor No odor	 No odor No odor
	Sodium percarbonate 5 wt.% 5 wt.%	Glucose tetraacetate 5 wt.% 5 wt.%	Magnesium sulfate 49 wt.% 9 wt.%	Sodium sulfate 40 wt.% 80 wt.%	Citric acid 1 wt.% 1 wt.%	 No odor No odor	 No odor No odor
	Sodium perborate 5 wt.% 5 wt.%	Sucrose hexaacetate 5 wt.% 5 wt.%	Magnesium sulfate 49 wt.% 9 wt.%	Sodium bicarbo-nate 40 wt.% 80 wt.%	Citric acid 1 wt.% 1 wt.%	 No odor No odor	 No odor No odor
Comparative product	Sodium percarbonate 5 wt.%	Glycerin diacetate 5 wt.%	Magnesium sulfate 49 wt.%	0 wt.%	Water 41 wt.%	Weak acetic acid odor	Weak acetic acid odor
	5 wt.%	5 wt.%	9 wt.%	0 wt.%	81 wt.%	Weak acetic acid odor	Weak acetic acid odor

Example 3

The sterilizing power of the composition according to the present invention was studied against *Escherichia Coli* IFO 3796 by modifying the Tanaka's method [Tokumitsu Tanaka, "Method for Measuring Sensitivity to Chemicals" edited by Susumu Mitsuhashi, Kodansha, Tokyo Japan (1980)].

Described specifically, about 100 μℓ (±5%) of precultured cells (about 10^8 cells/mℓ) were taken. They were inoculated to a test tube containing 10 mℓ of a solution of an invention germicidal composition which solution had been prepared 15 minutes ago by diluting the composition a predetermined number of times in distilled sterile water, and the solution was allowed to act on them at room temperature. At fixed intervals after the inoculation, the contents of the test tube were sampled out by a platinum loop and inoculated to a 96-well Petri dish (product of Corning Glass Works.; 370 μℓ per well) whose wells each contained 300 μℓ of a postculture medium. After the cells were incubated at 37°C for 2 days, the growth of the cells was determined. Incidentally, all the compositions shown in Tables 5 to 7 contained, in addition to the components (A), (B) and (C), 1% of citric acid, the balance being anhydrous sodium sulfate.

As is presented in Tables 5 to 7, it should be understood that each composition according to the present invention has excellent sterilizing effects.

Table 5

	(A) Inorganic peroxide	(B) Activator for the inorganic peroxide	(C) Alkali earth metal salt	(A)/(B)/(C), (wt.%)	Dilution, in terms of times, at which E. coli can survive 5-min contact but not 15-min contact
Invention product	Sodium percarbonate	Glycerin monoacetate (esterification degree: 33.3%)	Magnesium sulfate	5/0/0 5/5/0 5/5/5 5/5/25 5/5/40 5/5/75 5/0/40 0/100/800	X 500 X 1000 X 5000 X 10000 X 50000 X 50000 < X 1000 X 100 <
Invention product	Sodium percarbonate	Glycerin diacetate (esterification degree: 66.7%)	Magnesium sulfate	5/0/0 5/5/0 5/5/5 5/5/25 5/5/40 5/5/75 5/0/40 0/5/40	X 500 X 1000 X 5000 X 10000 X 50000 X 50000 X 1000 X 100 <
Comparative product	Sodium percarbonate	Glycerin triacetate (esterification degree: 100%)	Magnesium sulfate	5/0/0 5/5/0 5/5/5 5/5/25 5/5/40 5/5/75 5/0/40 0/5/40	X 500 X 200 X 1000 X 5000 X 5000 X 1000 X 100<

Table 6

	(A) Inorganic peroxide	(B) Activator for the inorganic peroxide	(C) Alkali earth metal salt	(A)/(B)/(C), (wt.%)	Dilution, in terms of times, at which E. coli can survive 5-min contact but not 15-min contact
Invention product	Sodium percarbonate	Glucose triacetate (esterification degree: 60%)	Magnesium sulfate	5/0/0	X 500
				5/5/0	X 2000
				5/5/5	X 5000
				5/5/25	X 50000
				5/5/40	X 50000 <
				5/5/75	X 50000 <
				5/0/40	X 1000
				0/5/40	X 100 <
	Sodium percarbonate	Glucose tetraacetate (esterification degree: 80%)	Magnesium sulfate	5/0/0	X 500
				5/5/0	X 2000
				5/5/5	X 5000
				5/5/25	X 50000
				5/5/40	X 50000 <
				5/5/75	X 50000 <
				5/0/40	X 1000
				0/5/40	X 100 <
Comparative product	Sodium percarbonate	Glycose pentaacetate (esterification degree: 100%)	Magnesium sulfate	5/0/0	X 500
				5/5/0	X 500
				5/5/5	X 1000
				5/5/25	X 2000
				5/5/40	X 5000
				5/5/75	X 5000
				5/0/40	X 1000
				0/5/40	X 100 <

Table 7

	(A) Inorgani peroxid	(B) Activator for the inorganic peroxide	(C) Alkali earth metal salt	(A)/(B)/(C), (wt. %)	Dilution, in terms of times, at which E. coli can survive 5-min contact but not 15-min contact
Invention product	Sodium perborate	Sucrose tetraacetate (esterification degree: 50%)	Magnesium sulfate	5/0/0 5/5/0 5/5/5 5/5/25 5/5/40 5/5/75 5/0/40 0/100/800	X 500 X 1000 X 5000 X 10000 X 10000 X 20000 X 1000 X 100 <
	Sodium perborate	Sucrose hexaacetate (esterification degree: 75%)	Magnesium sulfate	5/0/0 5/5/0 5/5/5 5/5/25 5/5/40 5/5/75 5/0/40 0/5/40	X 500 X 1000 X 5000 X 10000 X 10000 X 20000 X 1000 X 100 <
Comparative product	Sodium perborate	Sucrose octaacetate (esterification degree: 100%)	Magnesium sulfate	5/0/0 5/5/0 5/5/5 5/5/25 5/5/40 5/5/75 5/0/40 0/5/40	X 500 X 500 X 750 X 1000 X 1000 X 1000 X 1000 X 100 <

Industrial Applicability

The germicidal compositions according to the present invention have strong and long-lasting sterilizing effects and also excellent storage stability, so that they can be employed at a wide variety of places ranging from various factories to home.

CLAIMS

1. A germicidal composition comprising the following components (A), (B) and (C):

(A) an inorganic peroxide,

(B) an incomplete ester of a polyhydric alcohol with an organic acid, and

(C) an alkali earth metal salt.

2. A germicidal composition of claim 1, wherein the polyhydric alcohol is at least one of glycerins and sucroses.

3. A germicidal composition of claim 1 or 2, wherein the incomplete ester is an incomplete ester of a C_{1-8} fatty acid.

4. A germicidal composition of claim 1, 2 or 3, wherein the incomplete ester has an esterification degree of 5-95%.

5. A germicidal composition of claim 1, 2, 3 or 4, which comprises, based on 1 part by weight of the component (A), 0.01-10 parts by weight of the component (B) and 0.01-90 parts by weight of the component (C).

6. A germicidal composition of claim 1, further comprising an alkali metal salt.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/JP 94/00710

A. CLASSIFICATION OF SUBJECT MATTER
IPC 5 A01N59/00 A01N37/02 //(A01N59/00,37:02)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 5 A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X,Y	JP,A,62 155 203 (EISAI KK & KAO CORP.) 10 July 1987 see abstract	1-6
X	GB,A,1 496 856 (KAO SOAP COMPANY) 5 January 1978 see page 1, lines 47-95 and page 2, lines10 - 40.	1-6
Y	EP,A,0 047 015 (RICHARDSON-VICKS PTY. LTD.) 10 March 1982 see page 5, paragraph 1	1-6
A	US,A,4 051 059 (HENKEL & CIE. GMBH) 27 September 1977 cited in the application see column 1, line 30 - line 49	1-6

☐ Further documents are listed in the continuation of box C.

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